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ANNUAL TECHNICAL REPORT
NANOMECHANICS OF THIN FILMS

DEPARTMENT OF PHYSICS
CASE WESTERN RESERVE UNIVERSITY

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STATEMENT "A" per Dr. R. Brandt
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JUNE, 1990



Our work on the nanomechanics of thin films and microfibrils and interfaces is proceeding in a three-pronged approach. Each area is described briefly but only the highlights are given. Where an abstract exists it will be quoted. The final report of the entire effort will be issued in December, 1990.

During the second year of this research we have been concentrating on deposited films and interfaces. The goal during this time period was to examine the structure property relations for mechanical properties in aluminum and aluminum oxide multilayers. Briefly the aluminum films have been deposited in various thicknesses in UHV by evaporation from a W source and subsequently examined by AES, TEM and other surface analyses. For the mechanical data free standing aluminum films were formed by stripping in water from Victawer[®] edge coated thin glass slides following our earlier published techniques.[1] Highlights of the results are as follows:

A. Surface Analysis and Direct Mechanical Measurements

The free standing films are mounted without using glues on the nano-tensilometer with a typical stress strain curve as in reference 2. Note that the elastic modulus is that corresponding to bulk aluminum material and expected effect reachable only with some difficulty as described below. Linear elasticity is found only over a narrow strain range. Non-linear and irreversible behavior in small amounts is commonly found. A nominal stress strain curve shows only limited strain hardening behavior. Correlations between grain size and thickness and grain size and flow stress are noted. The films fail by intergranular fracture at strains of approximately 1-2% and attempts to identify impurities at grain boundaries by high spatial resolution SAM should be successful by contract end. Transparent amorphous Al₂O₃ films have been produced by depositions in low partial pressures of oxygen so that

interfaces may be constructed. This work should also be completed by 1 January 1991. Conventional wisdom now suggests that strong interfacial effects from superlattices are not to be anticipated. We reproduce some material from a poster paper presented at the NorthCoast Chapter of the AVS in late May, 1990. The abstract follows and figure 1 represents the production of aluminum films. Figure 2 from that poster shows the grain size thickness dependence. Additional details will be found in Ref. 1 and 2.

Abstract for NorthCoast AVS Symposium

28 May, 1990

Cleveland, Ohio

Thin aluminum films were deposited at room temperature by physical vapor deposition in ultra-high vacuum for mechanical testing. The film thickness ranged from 20 to 600 nm. Auger electron spectroscopy (AES) showed pure aluminum in the bulk, and a naturally formed layer of amorphous Al_2O_3 , with a thickness of 3 to 5 nm, on the surface. Transmission electron spectroscopy (TEM) of these specimens showed the average grain size to vary proportionally with thickness.

The samples ranged from 2 to 5 mm in length and 150 μm in width. These Al films, like most deposited films, exhibited brittle behavior when strained uniaxially until fracture. It is also observed that the failure is intergranular. In an attempt to identify the mode of embrittlement, AES was used to investigate impurities at grain boundaries and the fracture edge.

This work was supported by the Office of Naval Research through N0014-89-J-1555.

B. Finite Element Mechanics

In order to determine whether a measured Young's modulus for a film strained in uniaxial tension gave the same value as would be anticipated from bulk wire measurements, finite element analysis was required. As a result, limits have been set on sample size and mounting in order to allow for a use

of uniaxial straining to determine Young's modulus. The earlier two-dimensional calculations [3] have been supplanted by using a three-dimensional analysis. We have found that a dimensional ratio of approximately 5-1 for a length to width and mounting of the specimen without gues are necessary.

The abstract of a paper submitted to the AVS meeting in Toronto in October is attached and figures 3 and 4 indicate some of the stress contours found from our modelling.

C. Discontinuous Gold Films

EXAFS of strained gold thin films has been determined by electron detection using beam line X-11 at NSLS. Owing to the nature of synchrotron radiation work we have had one highly successful, one no data run and one run in May, 1990 which was partially successful but not yet analyzed. The objective of this work is to obtain strain measurements directly from specimens which are in the isolated island stage of growth. Discontinuous films that are not self supporting and hence, cannot be directly measured. Gold films have been prepared on plastic substrates in the thickness range from approximately 15 Å to 100 Å. Edge data has been obtained but reliable EXAFS data is yet to be determined in fall, 1990 runs.

The total effort under this contract is too substantial to allow completion of all of the results anticipated. The graduate students involved in this research are, K. Chaffee, G DeRose, D. Krus and G. Mearini. As a result, we have concentrated on the direct mechanical behavior and acknowledge with appreciation the contributions of the high resolution SAM at NASA LeRC. For further information please contact R.W. Hoffman, (216)368-4012.

References

1. R.W. Hoffman, Mat. Res. Soc. Symp. Proc. 69, 95 (1986).
2. R.W. Hoffman, Mat. Res. Soc. Symp. Proc. 130, 295 (1989).
3. D. Krus and R. Mullen in Ref. 2.

PRODUCTION OF ALUMINUM FILMS

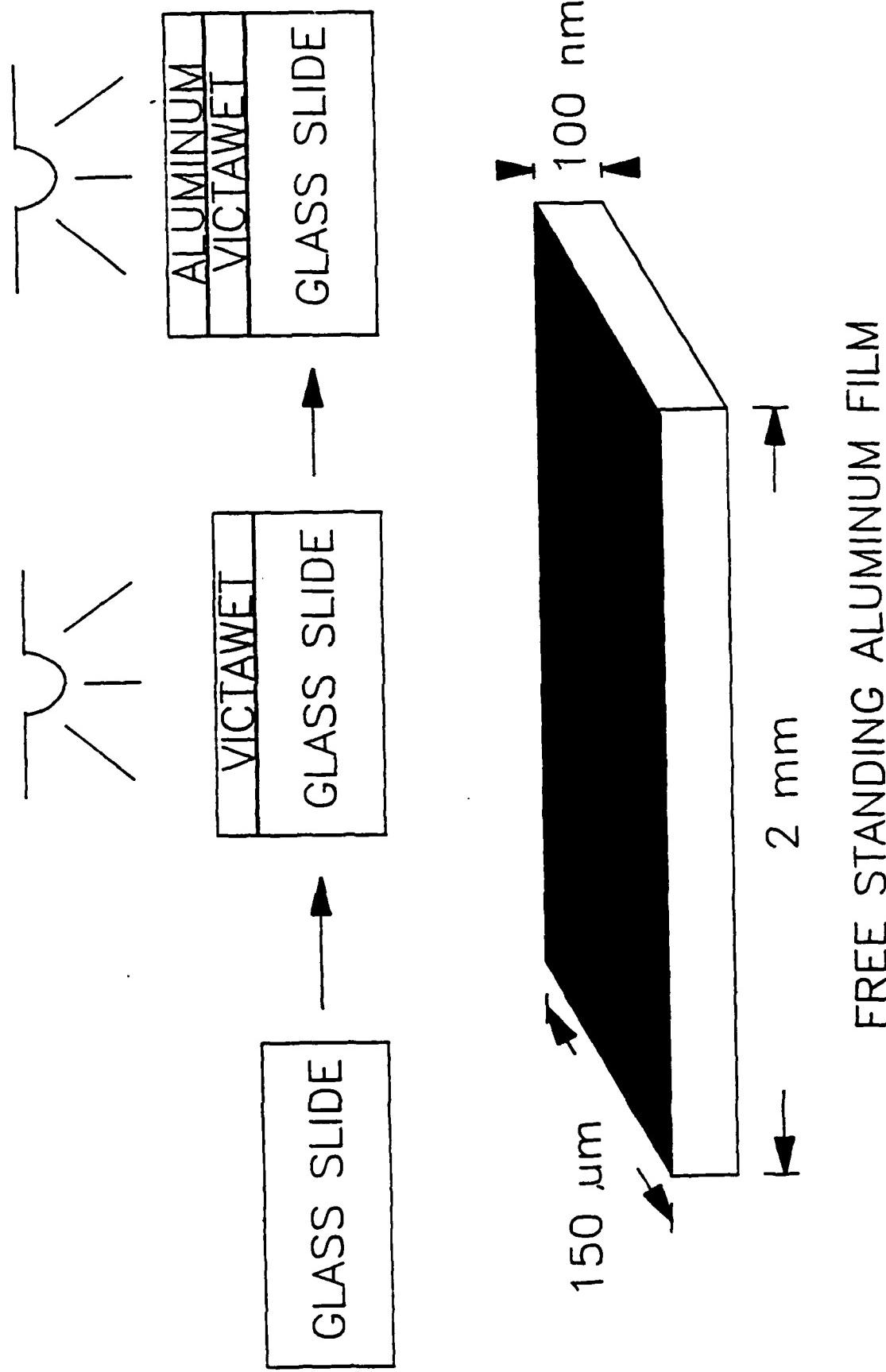


FIGURE 1 SAMPLE PREPARATION

Average Grain Diameter vs Film Thickness for PVD Al Films at Room Temperature

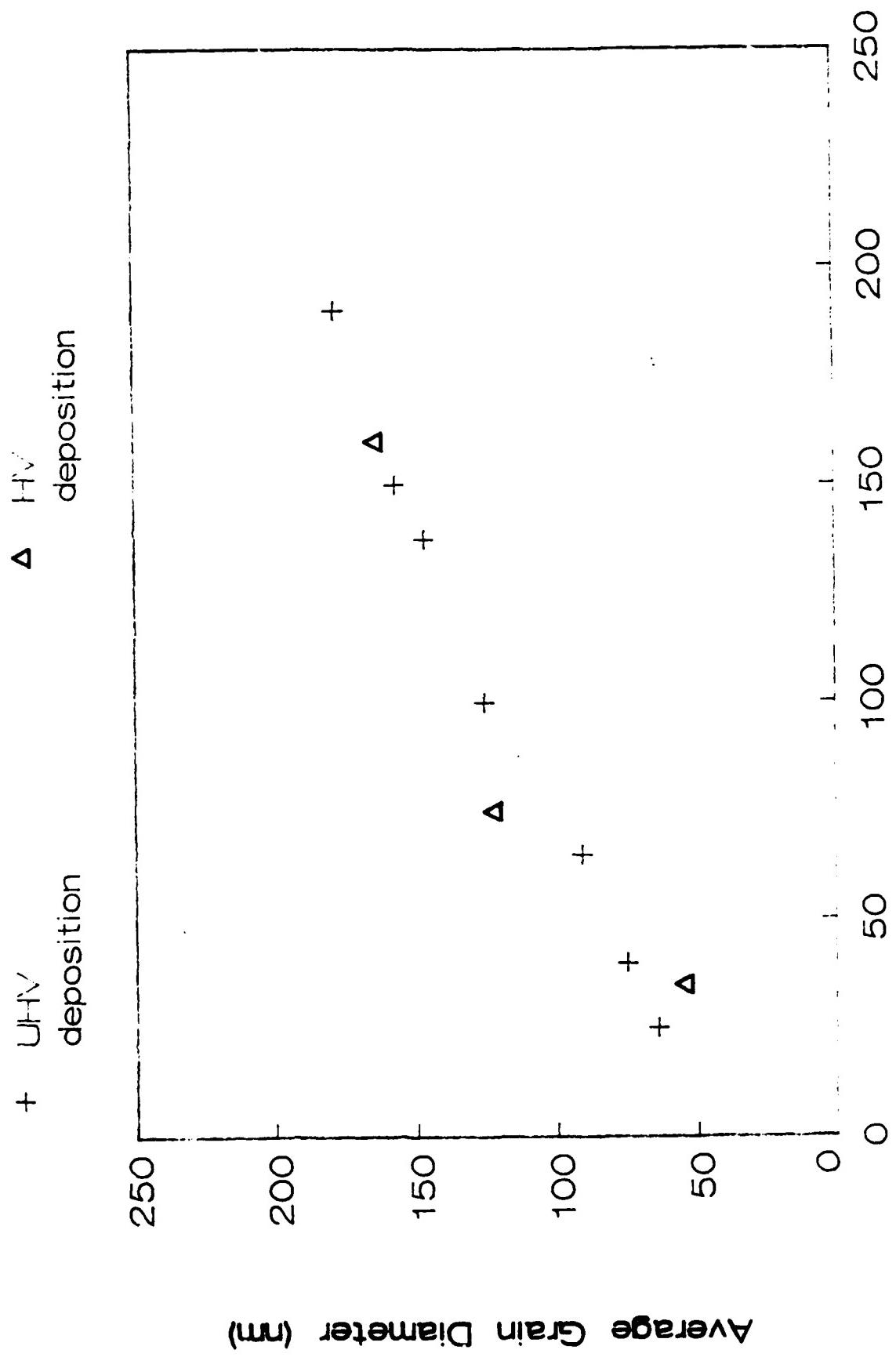


FIGURE 2 ALUMINUM FILM - GRAIN SIZE DEPENDENCE

Abstract Form

37th Annual AVS Symposium & Topical Conferences

Abstract Deadline is Friday, May 11, 1990, 5pm EDST

SESSION CODE

Start First Line Here

Underline or bold Title and Speaker's Name

TC1

OF THIN FILM STRESSES USING ALGOR PC BASED SOFTWARE. D.KRUS, JR., G.MEARINI, K.CHAFFEE, R.W.HOFFMAN. CASE WESTERN RESERVE UNIVERSITY, CLEVELAND, OH 44106.

Algor, a commercially available, PC based, finite element analysis software package was used to study three dimensional stress-strain relations in both free standing and adherent thin films. The accuracy of the analyses was verified by comparison to tensile tests and analytical radius of curvature calculations. The results also agree with earlier two dimensional calculations.

For free standing films, the program was used to model tensile testing in order to establish limits on film dimensions to yield Young's Modulus upon the application of a uniaxial stress approximation. Stress contours and submicroscopic elastic Poisson effects (necking) were determined for different film dimensions. In addition, the effect of using adhesives in sample mounting for tensile testing was studied, with the three dimensional model and results compared to similar two dimensional calculations.

For adherent films, stress distributions at film-substrate interfaces were determined for films in either tension or compression. Various geometries were considered, including films deposited around substrate corners. Internal cross sections as well as outer surfaces were used to determine the relation of sample geometry to the shapes and magnitudes of stress contours.

Supported by ONR through Contract # N0014-89-J-1555

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If you are submitting the abstract to one of the TOPICAL CONFERENCES, enter here the session code you would prefer as an alternative in the Annual Symposium:

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Is the Corresponding Author an AVS member? _____
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(2) D.W. Hoffman

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(3) T. Evans

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1000A Thick Al Film
Free-standing Film

0.2% Strain (tension)
1.2mm X 0.9mm

Sig xx ($\times 10^{-6}$ GPa)

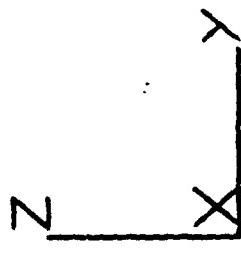
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FIGURE 3 UNIAXIAL STRESS CONTOURS - FEM

CORN2D6 . Minimum Principal Stress

Al203 (0.2 microns) on Al (2.0 microns square)



Strain=0.1% Compression

$\times 10^6$ = GPa

H	.00000
-0	.0046
-0	.0093
-0	.0140
-0	.0186
-0	.0233
L	.0280

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FIGURE 4 PRINCIPAL STRESS CONTOURS - FEM